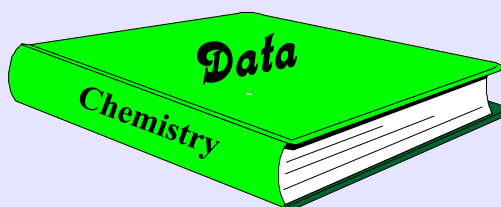


Chemistry Practical Work

Part III: Useful Information & Data

Safety Rules
Elements
Organic Compounds
Indicators
Laboratory reagents
Chromatography separations



Safety Rules for a Laboratory in which Chemistry Practical Work is performed.

1. Safety glasses or goggles must always be worn in a laboratory in which chemistry practical work is being performed. They are particularly important when corrosive chemicals are being handled and when liquids and solids are being heated in test-tubes.
2. Tie hair back to provide clear vision and avoid contact with naked flames.
3. Wear a laboratory coat to help protect skin and clothing from contact with chemicals.
4. Complete a risk assessment before you start practical work.
5. Substances releasing dangerous concentrations of toxic vapours must be handled in a fume-hood/cupboard.
6. Pipettes must be filled safely using pipette fillers (**Care:** If you are unsure how to use the filler safely ask the laboratory technician or lecturer-in-charge). Burettes must be filled safely keeping the top of the burette below eye level when pouring liquid into the burette (this is to avoid splashes in the eye).
7. Labels must be read very, very carefully before a reagent is used.
8. All glassware must be checked for cracks and broken edges before use, and returned to the technician if defective.
9. Buchner flasks must only be used when clamped in a suitable stand.
10. Chemicals must never be returned to reagent bottles (small, excess, quantities of aqueous solutions can be washed away down the sink).
11. Coats and general personal belongings are not to be kept on work benches or on the floor where, during a practical session, they will pose a potential hazard (do not cover fire-extinguishers with coats, etc).
12. All spillages are to be cleaned up immediately and completely. When the laboratory is left, each student must check that their work area is clean and nothing has been left in sinks.
13. Mains leads on electrical equipment must not be tampered with, any defective wiring must be reported immediately.
14. Smoking, eating and drinking are not permitted in the laboratory.

**Part III: Data
Elements:**

Name	Symbol	Atomic number	Relative Atomic Mass	Mpt °C	Bpt °C
Hydrogen	H	1	1	-259	-253
Helium	He	2	4	-270	-269
Lithium	Li	3	6.9	181	1331
Beryllium	Be	4	9	1283	2477
Boron	B	5	10.8	2027	3927
Carbon	C	6	12	3727 diamond	4800 diamond
Nitrogen	N	7	14	-210	-196
Oxygen	O	8	16	-219	-183
Fluorine	F	9	19	-220	-188
Neon	Ne	10	20.2	-248	-246
Sodium	Na	11	23	98	890
Magnesium	Mg	12	24.2	650	1117
Aluminium	Al	13	27	659	2447
Silicon	Si	14	28.1	1410	2677
Phosphorus	P	15	31	44 white	281 white
Sulphur	S	16	32.1	113 rhombic	445 rhombic
Chlorine	Cl	17	35.5	-101	-34
Argon	Ar	18	39.9	-189	-186
Potassium	K	19	39.1	63	766

Elements:

Name	Symbol	Atomic number	Relative Atomic Mass	Mpt °C	Bpt °C
Calcium	Ca	20	40.1	850	1487
Scandium	Sc	21	45	1540	2730
Titanium	Ti	22	47.9	1677	3277
Vanadium	V	23	50.9	1916	3360
Chromium	Cr	24	52	1903	2642
Manganese	Mn	25	54.9	1244	2041
Iron	Fe	26	56	1538	2890
Cobalt	Co	27	58.9	1492	2880
Nickel	Ni	28	58.7	1728	3110
Copper	Cu	29	63.5	1083	2595
Zinc	Zn	30	65.4	420	907
Gallium	Ga	31	69.7	30	2300
Germanium	Ge	32	72.6	937	2830
Arsenic	As	33	74.9	820 At 37 atmos- pheres pressure	630 sublimes
Selenium	Se	34	79	217	685
Bromine	Br	35	80	-7	58
Krypton	Kr	36	83.8	-157	-153
Rubidium	Rb	37	85.5	39	701
Strontium	Sr	38	87.6	769	1370

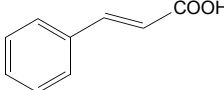
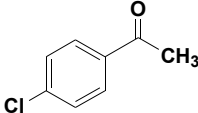
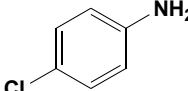
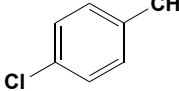
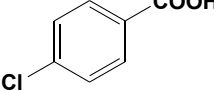
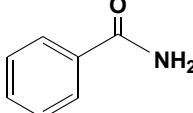
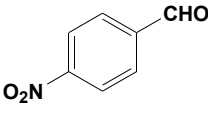
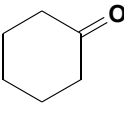
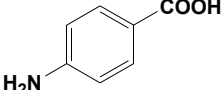
Organic Compounds: (some of these *may* be considered suitable for use in organic analysis)

Name	Molecular Formula	Relative molecular mass	%C	%H	%O	%N, Cl, Br, S	Mpt /bpt °C	Derivative Mpt °C	code
cinnamic acid	C ₉ H ₈ O ₂	148	72.97	5.40	21.62	0.00	133	Amide 141	1
p-chloroacetophenone	C ₈ H ₇ OCl	154.5	62.13	4.53	10.36	22.97	bpt 232	2,4DNP 236	2
p-chloroaniline	C ₆ H ₆ NCl	113.5	56.47	4.71	0.00	N=10.98 Cl=27.84	70 bpt 230	benzoyl 192	3
p-chlorobenzaldehyde	C ₇ H ₅ OCl	140.5	59.78	3.56	11.33	25.27	47	2,4DNP=265 semicarbazone=232	4
p-chlorobenzoic acid	C ₇ H ₅ O ₂ Cl	156.5	53.67	3.19	20.44	22.68	243	amide=179	5
benzamide	C ₇ H ₇ ON	121	69.42	5.79	13.22	11.57	129	benzoic acid=121	6
p-nitrobenzaldehyde	C ₇ H ₅ O ₃ N	151	55.63	3.31	31.79	9.27	106	2,4DNP=320 semicarbazone=221	7
cyclohexanone	C ₆ H ₁₀ O	98	73.46	10.20	16.33	0.00	bpt 155	2,4DNP=162 semicarbazone=166	8
p-aminobenzoic acid	C ₇ H ₇ O ₂ N	137	61.32	5.11	23.35	10.22	188	benzoyl 278	9
p-nitrobenzamide	C ₇ H ₆ O ₃ N ₂	166	50.61	3.62	28.91	16.86	200	nitrobenzoic acid 241	10
sulcatone 6-methylhept-5-en-2-one	C ₈ H ₁₄ O	126	76.20	11.10	12.70	0.00	bpt 174	semicarbazone 131	11
p-hydroxyacetophenone	C ₈ H ₈ O ₂	136	70.59	5.88	23.53	0.00	110	2,4DNP 261 semicarbazone 199	12
benzyl alcohol	C ₇ H ₈ O	108	77.78	7.41	14.81	0.00	bpt 205	3,5-dinitrobenzoate 113	13
o-nitrobenzaldehyde	C ₇ H ₅ O ₃ N	151	55.60	3.30	31.80	9.30	44	2,4DNP 265	14
propiophenone	C ₉ H ₁₀ O	134	80.60	7.50	11.90	0.00	bpt 218	2,4DNP 191 semicarbazone 174	15
benzophenone	C ₁₃ H ₁₀ O	182	85.70	5.50	8.80	0.00	49	2,4DNP 238 semicarbazone 165	16

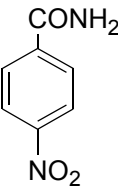
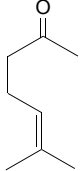
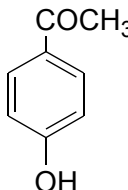
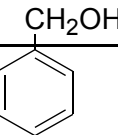
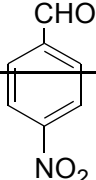
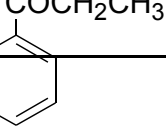
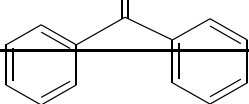
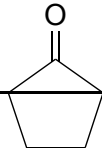
Organic Compounds: (continued)

Name	Molecular Formula	Relative molecular mass	%C	%H	%O	%N, Cl, Br, S	Mpt /bpt °C	Derivative Mpt °C	code
cyclopentanone	C ₅ H ₈ O	84	71.43	9.52	19.05	0.00	bpt 131	2,4DNP 146 semicarbazone 206	17
benzanilide	C ₁₃ H ₁₁ ON	197	79.20	5.60	8.10	7.10	161	benzoic acid 121	18
salicylic acid	C ₇ H ₆ O ₃	138	60.90	4.30	34.80	0.00	159	5-nitroderiv 226 amide 139	19
o-chlorobenzoic acid	C ₇ H ₅ O ₂ Cl	156.5	53.70	3.20	20.40	22.70	137	amide 139	20
succinimide	C ₄ H ₅ O ₂ N	99	48.48	5.05	32.32	14.14	125	succinic acid 185	21
succinamide	C ₄ H ₈ O ₂ N ₂	116	41.38	6.90	27.58	24.14	242	succinic acid 185	22
phenylalanine	C ₉ H ₁₁ O ₂ N	165	65.45	6.67	19.39	8.48	273	benzoyl 187	23
benzoin	C ₁₄ H ₁₂ O ₂	212	68.57	8.57	22.86	0.00	133	2,4DNP 245 semicarbazone 206 d	24
4-methylacetophenone	C ₉ H ₁₀ O	134	80.60	7.46	11.94	0.00	bpt 226	2,4DNP 260 semicarbazone 205	25
ethanol	C ₂ H ₅ O	45	53.33	11.11	35.56	0.00	bpt 78	ethylbenzoate bpt 213 3,5-dinitrobenzoate 92	26
butanal	C ₄ H ₈ O	72	66.67	11.11	22.22	0.00	bpt 74	2,4DNP 122 semicarbazone 126	27
butanone	C ₄ H ₈ O	72	66.67	11.11	22.22	0.00	bpt 80	2,4DNP 115 semicarbazone 135	28
ethanoic acid	C ₂ H ₄ O ₂	60	40.00	6.67	53.33	0.00	16 bpt 118	amide 82	29
benzylamine	C ₇ H ₉ N	107	78.50	8.41	0.00	13.08	bpt 185	benzoyl 105	30

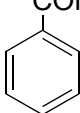
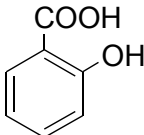
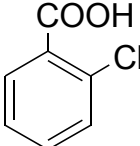
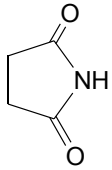
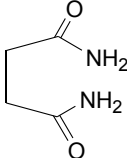
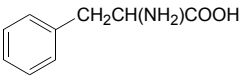
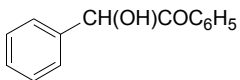
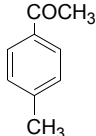
Organic compounds:

code	common name	systematic name	structure	Infra red absorptions Main features (cm^{-1}) (KBr or liquid film)	Pmr absorptions Main features (ppm) (in CDCl_3)
1	cinnamic acid	3-phenylpropenoic acid (E isomer)		3065, 3055 (ar-CH str) 2940, 2840 (ali-CH str) 1680 (conj CO str) 1624 (conjC=C str)	6.45 (d, unsat-H) 7.40(d, unsat-H) 7.55(d, arom-H) 7.80(d, arom-H) (carboxyl proton not observed)
2	p-chloroacetophenone	4'-chlorophenylethanone (or 1-chloro-4-ethanoylbenzene)		3100-3000 (arom-CH str) 1690 (conj CO str) 470 (arom C-Cl)	7.88 (complex doublet, arom-2H) 7.43 (complex doublet, arom-2H) 2.58 (s, aliph-3H)
3	p-chloroaniline	4-chlorobenzenamine		3420, 3350, 3210 (NH_2 str) 3040 (complex, arom-CH str)	7.15 (complex doublet, arom-2H) 6.55 (complex doublet, arom-2H) 3.60 (broad, s, N-2H)
4	p-chlorobenzaldehyde	4-chlorobenzenecarbaldehyde		3080 (arom-CH str) 2840, 2730 (ald-CH str) 1700 (ald-CO str)	10.00 (s, ald-H) 7.86 (d, arom-2H) 7.57 (d, arom-2H)
5	p-chlorobenzoic acid	4-chlorobenzenecarboxylic acid		Very broad and sloping absorption band between 3600 & 2700 (COOH). Superimposed on this spikes at 3200 (OH) & 3080 (C_6H_4). Large, broad, peak at 1695 (CO).	7.92 (d, arom-2H) 7.42 (d, arom-2H) carboxyl proton not showing (possibly very broad)
6	benzamide	benzenecarboxamide		3375 & 3180 (NH_2 str) 3060 (arom-CH) 1620 (CO str) 1625 (NH_2 bend) 1400 (NH_2 bend)	7.90 (d, arom-2H) 7.45 (complex, arom-3H) 7.20 (broad singlet NH_2)
7	p-nitrobenzaldehyde	4-nitrobenzenecarbaldehyde		3107 (arom-H) 2862 (ald-H) 1710 (ald-CO) 1680 (arom-H) 1543 (arom- NO_2) 815 (arom- NO_2)	10.20 (ald-H) 8.40 (d, arom-2H) 8.12 (d, arom-2H)
8	cyclohexanone	cyclohexanone		3400 (CO overtone) 2900 (strong aliph-CH str) 1715 (strong CO str)	2.35 (t, 4H) 1.84 (complex, 4H) 1.70 (complex, 2H)
9	p-aminobenzoic acid	4-aminobenzenecarboxylic acid		3470 (arom- NH_2 str) 3370 (arom- NH_2 str) 3200-2500 (broad COOH) 1680 (carboxyl CO str)	7.73 (d, arom-2H) 6.60 (d, arom-2H) carboxyl proton not showing (possibly very broad)


Organic compounds (continued):

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10	p-nitrobenzamide	4-nitrobenzene-carboxamide		3479, 3182 (amide NH ₂ str) 3121 (complex arom-4H str) 1680 (amide CO str) 1604 (amide, NH ₂) 1528, 1347 (NO ₂ str)	Predicted features: 8.47 (d, arom-2H) 7.90 (d, arom-2H) 7.00 (broad singlet NH ₂)
11	sulcatone	6-methylhept-5-en-2-one		2800-2950 (complex, sat CH ₂ /CH ₃) 1700 (CO str)	5.02 (complex, C=C-H) 2.32 (complex, 2CH ₂) 2.10 (CH ₃ CO) 1.61 (d, 2CH ₃)
12	p-hydroxyacetophenone	4'-hydroxyphenylethanone		3300-2800 (broad intense absorption with superimposed peaks) 3300 (OH str) 2900 (complex, CH ₃ str) 1680 (CO str) 1280, 1200 (C-O, OH) 1580 (arom-H bend)	7.93 (d, arom-2H) 6.99 (d, arom-2H) 2.60 (s, CH ₃)
13	benzyl alcohol	phenylmethanol		3300 (broad intense OH) 3100-3020 (arom-H str) 2900 (CH ₂ str) 1050-1000 (C-O, CH ₂ OH)	7.25 (complex, arom-5H) 4.50 (s, CH ₂) 3.30 (complex, OH)
14	o-nitrobenzaldehyde	2-nitrobenzaldehyde		3105 (arom-H str) 2854 (ald-CH str) 1699 (ald-CO) 1570 (arom C=C) 1532, 1346 (arom-NO ₂)	10.42 (s, ald-H) 8.42-7.78 (complex, arom-4H)
15	propiophenone (hawthorne ketone)	1-phenylpropanone		3370 (CO overtone) 3080 (complex, arom-H str) 2920 (complex, CH ₂ /CH ₃ str) 1685 (CO str)	7.95 (d, arom-2H) 7.50 (complex, arom-3H) 2.98 (quartet, CH ₂) 1.20 (t, CH ₃)
16	benzophenone	diphenylmethanone		3070 (complex, arom-H str) 1655 (s, conj CO str) 1600, 1580 (two s, arom-C=C str) 1280 (s, C=C bend) 770, 700 (C ₆ H ₅ -group)	7.80 (d, arom-2H) 7.55 (complex d, arom-3H)
17	cyclopentanone	cyclopentanone		2975-2880 (complex, CH ₂ str) 1745 (CO str)	2.16 (d, 2CH ₂) 1.97 (d, 2CH ₂)

Organic compounds (continued):

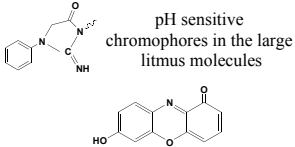
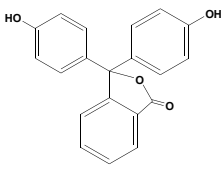
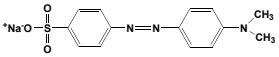
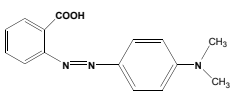
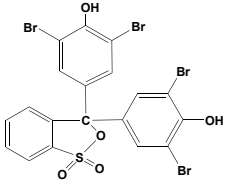
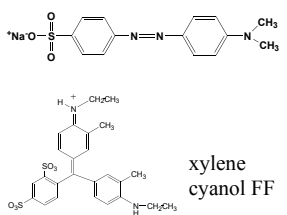
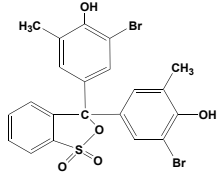
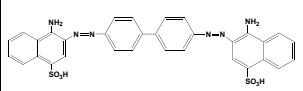
code	common name	systematic name	structure	Infra red absorptions Main features (cm ⁻¹)	Pmr absorptions Main features (ppm)
18	benzanilide	N-phenylbenzenecarboxamide		3345 (NH str) 3070 (complex, arom-H) 1660 (amide CO)	10.28 (NH) 7.90-7.10 (complex arom-10H)
19	salicylic acid	2-hydroxybenzene carboxylic acid		3013-2605 (typical broad absorption due to COOH) 3240 (OH) 3013 (arom-H str) 1662 (carboxyl-CO str) 1613 (arom C=C str)	12-11 (broad, H-bonded OH protons) 7.75 (complex, arom-1H) 7.45 (complex, arom-1H) 6.80 (arom-2H)
20	o-chlorobenzoic acid	2-chlorobenzene carboxylic acid		3200-2600 (broad, typical COOH) 3100 (complex, arom-H) 1690 (CO str) 1595, 1575 (arom C=C str) 745 (arom ortho substitution)	9.00 (broad, COOH) 7.80 (complex, arom-1H) 7.35 (complex, arom-3H)
21	succinimide	succinimide		3165, 3080 (NH str) 2950-2850 (CH ₂ str) 1780 -1700 (ring sys CONHCO)	11.10 (CONHCO) 2.60 (s, 2CH ₂)
22	succinamide	butane-1,4-di-amide		3340, 3175 (NH str) 2955-2850 (CH ₂ str) 1640, 1680 (2CONH ₂ sys)	Predicted: 11.00 (N-4H) 2.50 (2CH ₂)
23	phenylalanine	2-amino-3-phenylpropanoic acid		3100-2800 (broad, typical of amino acid) 3500 (complex, arom-H) 2950 (complex, sat C-H str) 1560 (carboxyl CO str)	7.30 (complex, arom-5H) 5.45 (s, D-O-H) 3.65 (complex) 3.20 (split doublet) 2.85 (complex)
24	benzoin	1,2-diphenyl-2-hydroxyethanone		3420, 3380 (OH) 3050 (complex, arom-H) 1686 (CO str) 1595, 1585 (arom C=C)	7.90 (d,) 7.50 (complex, arom-H) 7.30 (complex, arom-H) 5.95 (d,) 4.55 (d,)
25	4-methylacetophenone	1-ethanoyl-4-methylbenzene		3050 (complex, arom-H str) 2950 (complex, sat CH ₃) 1695 (CO str)	Predicted: 7.50 (typical, para substitution pattern) 2.60 (s, CH ₃ CO) 2.30 (s, CH ₃ -Ph)

Organic compounds (continued):

code	common name	systematic name	structure	Infra red absorptions Main features (cm ⁻¹)	Pmr absorptions Main features (ppm)
26	ethyl alcohol	ethanol	CH ₃ CH ₂ OH	3340 (O-H str) 2980-2850 (complex, CH ₃ /CH ₂) 1090, 1050 (O-H & C-O bend)	4.50 (broad s, OH) 3.75 (q, CH ₂) 1.25 (t, CH ₃)
27	butyraldehyde	butanal	CH ₃ CH ₂ CH ₂ CHO	2950 (complex, CH ₃ /CH ₂ str) 2720 (aldehyde CH str) 1700 (carbonyl str)	9.70 (triplet, CHO) 2.45 (triplet, CH ₂ -ald) 1.65 (complex, methylene-CH ₂ -methylene) 0.96 (triplet, CH ₃)
28	methyl ethyl ketone (MEK)	butanone	CH ₃ CH ₂ COCH ₃	2950 (complex, CH ₃ /CH ₂ str) 1730 (carbonyl str)	2.48 (quartet, CH ₂ -CO) 2.15 (singlet, CH ₃ -CO) 1.14 (triplet, CH ₃)
29	acetic acid	ethanoic acid	CH ₃ COOH	3400-2500 (intense, broad, COOH) 2700 (complex, CH ₃) 1740 (intense, broad, CO str)	11.40 (quartet, COOH) 2.05 (doublet, CH ₃)
30	benzylamine	phenylmethan-amine	 CH ₂ NH ₂	3155 (2 peaks, NH ₂ str) 3040 (complex, C ₆ H ₅) 2940 (complex, CH ₂)	7.30 (complex, C ₆ H ₅ -) 3.80 (singlet, NH ₂) 1.40 (singlet, CH ₂ -N)

Acid-Base Indicators

(Note: most indicators are suitable for titrating a strong acid against a strong base due to large ΔpH at end point)

Name	Structure	Colour in Acid (dil. solutions @ room temp.)	Colour in Alkali (dil. solutions @ room temp.)	pH range (& suitability)	pK_{In}
litmus (azolitmin & erythrolitmin)	Complex natural material (from lichens)  pH sensitive chromophores in the large litmus molecules	red	blue	4-8 (unsuitable for acid-base titrations due to large pH range)	-
phenolphthalein		colourless	pink	8-10 (weak acid (eg, ethanoic) against a strong base)	9.3
methyl orange		red	orange	3-4.5 (strong acid against a weak base (eg, sodium carbonate, ammonia))	3.7
methyl red		red	yellow	4.5-6 (strong acid against a weak base)	5.1
bromophenol blue		yellow	blue	3-5 (strong acid against a weak base)	4.0
screened methyl orange (methyl orange + xylene cyanol FF (a green dye))	 xylene cyanol FF	purple	green	3-5 (strong acid against a weak base)	3.7
bromocresol purple		yellow	purple	5-7 (strong acid-strong base, weak base-strong acid)	6.3
congo red		violet	red	3-5 (strong acid against a weak base)	4.0

Other indicators:

Type of titration	Indicator	Colour at end point	limitations
Silver nitrate for estimating halides.	5% potassium chromate solution. Suitable for estimating chloride.	Brick red due to silver chromate	Can only be used in neutral solution
	The adsorption indicator dichlorofluorescein(0.1% in 70% ethanol) is suitable for estimating the halides.	Pink colour (light sensitive)	Can be used in weakly acid conditions (eg, pH 4 - 7)
	The adsorption indicator, eosin, is suitable for estimating bromide, iodide & thiocyanate but not chloride .	Magenta colour.	Can be used in more acid conditions (eg, pH 1-2)
Silver nitrate for estimating thiocyanate	10% iron(III) ammonium sulphate	Colourless to blood red	
Potassium permanganate for estimating reducing agents (eg, iron(II), oxalate)	Self indicating	Slight pink due to excess permanganate ion in dilute aqueous solution.	Used in dilute sulphuric acid conditions (do not use hydrochloric or nitric acids)
Sodium thiosulphate for estimating iodine.	Starch solution (about 0.4% in water)	Dark blue to colourless	Use in neutral or acidic conditions (the thiosulphate solution must be prepared using acid free water)
Potassium dichromate for estimating reducing agents (eg, iron(II))	N-phenylanthranilic acid (0.1% in dilute sodium carbonate solution).	Green to red-violet.	Can be used in the presence of chloride ion (unlike permanganate).
	0.35% aqueous barium diphenylamine sulphate	Deep green to violet (addition of phosphoric acid to the titration mixture helps to sharpen the colour change at the end point).	This is a good reliable indicator (use about 0.5 cm ³ of indicator solution per titration)
Complexometric titration: eg, EDTA (ethylene diamine tetraacetic acid) For estimating metal ions in aqueous solution. (eg, nickel(II))	Murexide (ammonium purpurate) (10% murexide ground up in dry KBr. Use about 0.05g of this mixture per titration) Eriochromae black T is another very useful indicator for use with EDTA.	Yellow to bluish violet	The titration is carried out, first under neutral conditions and then, near the endpoint, in alkaline conditions; making alkaline using concentrated ammonia solution.

Useful Laboratory reagents

Reagent	Concentration	Safety
Sodium hydroxide solution	2M (80g per dm ³ of distilled water)	Caustic to the skin (when making up, dissolve the solid gradually, allowing for cooling, in distilled water in an operational fume cupboard)
Ammonia solution	2M (100 cm ³ of concentrated (ie, 0.88 ammonia) made up to 1 dm ³ with distilled water).	Caustic (make up the solution in a fume cupboard since ammonia fumes are very pungent).
Hydrochloric acid	2M (200 cm ³ of concentrated acid made up to 1 dm ³ with distilled water)	Corrosive
Nitric acid	2M (128 cm ³ of concentrated acid made up to 1 dm ³ with distilled water)	Corrosive (the concentrated acid is highly corrosive)
Sulphuric acid	1M (56 cm ³ of concentrated acid made up to 1 dm ³ with distilled water)	Corrosive (the concentrated acid is highly corrosive . When diluting, always add acid gradually to water!)
Lime water (calcium hydroxide solution)	Saturated calcium hydroxide solution (about 0.025M): Nearly fill a clean Winchester bottle with distilled water. Add about 20g of calcium hydroxide (slaked lime). Stopper the bottle and invert it a few times to thoroughly mix the contents. Allow to stand overnight. Decant some of the clear liquid into a 250 cm ³ , labelled, reagent bottle.	Avoid breathing in particles of calcium hydroxide when making up the solution.
Ammonium chloride solution	2M (100g of ammonium chloride dissolved in 1 dm ³ of distilled water)	-
Bromine water	Saturated : Take a clean 250 cm ³ , labelled, reagent bottle and nearly fill with distilled water. Add a small amount of liquid bromine (CARE). Seal the bottle with a good fitting stopper and invert a few times to encourage the bromine to dissolve. Continue adding bromine until no more will dissolve.	Highly corrosive. Make up the solution in a fume cupboard so as to avoid breathing the bromine vapour. Wear plastic gloves and safety spectacles to avoid skin contact with the bromine. The saturated solution can be diluted with distilled water as required.
Silver nitrate solution	0.1M (17g dissolved in 1 dm ³ of distilled water)	Corrosive. Avoid skin contact; the solid and solution will burn and blacken the skin.

Reagent	Concentration	Safety
Iron(III) chloride solution	0.5M (23g of hydrated salt in 1 dm ³ of distilled water. To prevent hydrolysis add about 30 cm ³ of concentrated hydrochloric acid to each litre of water before dissolving the solid)	Corrosive solution. (Labelled the reagent bottle so as to indicate that the solution has been acidified with HCl)
Copper(II) sulphate solution	0.5M (124g of hydrated salt in 1 dm ³ of distilled water)	Poisonous.
Sodium carbonate solution	10% (100g of anhydrous salt in 1 dm ³ of distilled water).	Slightly alkaline.
Iodine solution	0.1M (12.7g iodine dissolved in a dm ³ of distilled water containing 30g of dissolved potassium iodide).	Stains the skin brown. (the brown colouration can be removed by rinsing with dilute sodium thiosulphate solution)
Potassium permanganate solution	0.02M (3.16g dissolved in 1 dm ³ of distilled water)	Stains skin brown.
Sodium thiosulphate solution	0.1M (24.81g of the pentahydrate dissolved in 1 dm ³ of distilled water) This solution will keep some time providing the container is well sealed and the solution kept free of acid.	-
Potassium dichromate solution	0.02M (5.882g dissolved in 1 dm ³ of distilled water)	Toxic
2,4-dinitrophenylhydrazine solution (Brady's reagent)	0.2M (dissolve 10g of the hydrazine in 20 cm ³ of concentrated sulphuric acid (CARE). In a fume cupboard, slowly and gradually, add with stirring and cooling, 180 cm ³ of methanol. Decant the solution from any undissolved hydrazine and to the solution slowly add, with stirring, 50 cm ³ of distilled water.	Toxic and corrosive. Avoid skin contact.
Hydrogen peroxide solution	2M ('20 volume' ie, about 60g hydrogen peroxide per dm ³))	Oxidising reagent
Fehling's reagent	Made up fresh by mixing equal volumes of Fehlings solutions 1 & 2 Fehling's 1: 0.28M copper(II) sulphate solution (34.6g of the pentahydrate dissolved in 500 cm ³ of distilled water (acidified with a few drops of 1M sulphuric acid)). Fehling's 2: dissolve 60g sodium hydroxide in about 300 cm ³ of distilled water (CARE). In this solution dissolve 173g of sodium potassium tartrate and add, with stirring, another 200 cm ³ of distilled water.	Toxic and corrosive

Reagent	Concentration	Safety
Sodium hypochlorite solution (sodium chlorate(I) solution)	12-14% available chlorine (buy from chemical supplier)	Caustic Chlorine is generated when it is treated with dil. HCl)
Barium chloride solution	0.1M (24.4g per dm ³)	Toxic
Potassium iodide solution	0.2M (33.2g per dm ³)	Harmful by mouth
Sodium hydrogencarbonate solution	Saturated	Harmful by mouth
Magnesium sulphate solution	0.1M (24.6g of the heptahydrate in 1 dm ³ of distilled water)	Harmful by mouth
Zinc sulphate solution	0.1M (28.7g of the heptahydrate in 1 dm ³ of distilled water)	Harmful by mouth
Calcium chloride solution	0.1M (14.7g of the dihydrate in a dm ³ of distilled water)	Harmful by mouth
Sodium chloride solution	0.1M (5.9g per dm ³ of distilled water)	Harmful by mouth
Potassium nitrate solution	0.1M (10.1g per dm ³ of distilled water)	Harmful by mouth
Sodium hydrogensulphate	0.1M (12.0g per dm ³ of distilled water)	Irritant
Lithium chloride solution	0.1M (4.2g per dm ³ of distilled water)	Toxic

Chromatography

Paper chromatography

1. Separation of amino acids.

0.01% amino acids made up in propan-2-ol: water (1:9).

Whatman paper No.1.

Spray reagent: 0.02M (0.36%) ninhydrin (2,2-Dihydroxyindane-1,3-dione) in propanone (acetone). Keep in fridge.

(**CARE**: ninhydrin is toxic and colours the skin blue)

Heat the sprayed chromatogram at about 120°C (in small oven or using a hair dryer set on hot; avoid breathing toxic fumes by heating under a fume hood).

2. Analysis of Chlorophyll

Chlorophyll extract (extract crushed plant material with propanone; dry and evaporate to small volume).

Whatman chromatography paper No.1.

Eluant: propanone: petroleum spirit (40-60) (1:9 by volume)

(On a piece of paper, 20 cm high, twelve coloured spots should be visible).

3. Separation of Transition Metal Cations.

Whatman chromatography paper.

0.4M solutions of, iron(III) chloride, copper(II) chloride and nickel(II) chloride. These solutions are made up in 2M hydrochloric acid. A mixture of all three (equal volumes of each).

Eluting solvent: propanone: concentrated hydrochloric acid: water (86: 6: 8 cm³).

Locating the cations: spot the chromatogram (the paper) with the individual cations and at least three separate spots of the mixture. After developing, cut the chromatogram so that you have each of the cations on separate strips of paper.

Similarly with the mixture, cut three strips each with the mixture on it.

Take two strips one with iron(III) on it and one with the mixture on it. Spray these two strips with 0.25M ammonium thiocyanate solution. The iron(III) shows as a dark red spot.

Repeat this procedure for nickel(II) spraying the strips with a 1% solution of dimethylglyoxime in ethanol. Nickel shows red.

Repeat for copper(II), spraying the strips with 0.88 ammonia solution (**CARE**: use in the fume cupboard). Copper shows blue.

Thin layer chromatography

The following are useful for demonstrating the technique:

1. Separation of p-hydroxybenzaldehyde and benzophenone

Tlc film: Silica gel, eg, Machery-Nagel Polygram Sil G/UV 254

Apply the samples as spots of 1% solutions in ethanol or ether.

Eluting solvent: petroleum spirit (40-60): ethoxyethane (ether) (1:1).

Spray reagent: 2,4-DNPH solution (Brady's reagent). Spray in well ventilated fume cupboard. Spots show yellow/orange.

R_f values: p-hydroxybenzaldehyde 0.35, benzophenone 0.80.

2. Separation of amino acids.

Tlc film: silica gel.

Amino acids: various. 0.01% solution made up in propan-2-ol: water (1:9).

Eluting solvent: butan-1-ol: ethanoic acid (glacial acetic): water (12:3:5).

Spray reagent: 0.02M ninhydrin (1,2,3-indanetrione monohydrate) in propanone (keep in fridge).

Heat developed chromatogram at about 120°C (in small oven in the fume cupboard). Purple-blue spots.

R_f values: eg, lysine 0.07, aspartic 0.14, alanine 0.17, valine 0.28, leucine 0.40.

(Another useful eluting solvent for amino acids is, ethyl ethanoate: formic acid (90%): water (7:2:1 by volume)).

3. Separation of sugars.

Tlc film: silica gel.

Eluting solvent: propanone: butan-1-ol: water (5:4:1 by vol)

Spray reagent: alkaline silver nitrate solution (**CARE**: corrosive. Spray in a well ventilated fume cupboard.). Heat plate at about 120°C. The sugars appear as brown spots.

~R_f values: eg, maltose 0.22, lactose 0.30, glucose 0.40, galactose 0.50.

4. Separation of Dyes.

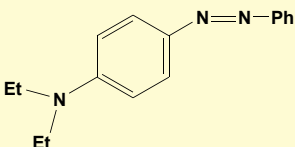
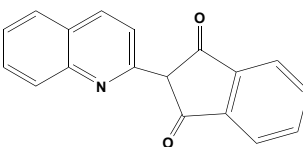
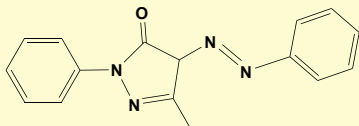
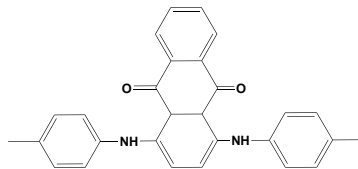
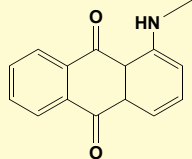
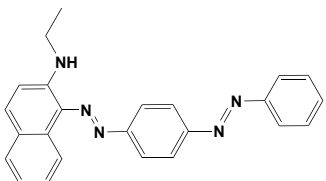
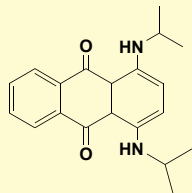
Tlc film: silica gel.

Eluting (& solution) solvent: *compare* dichloromethane and methylbenzene (toluene).

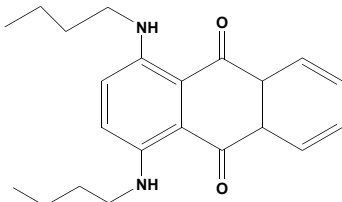
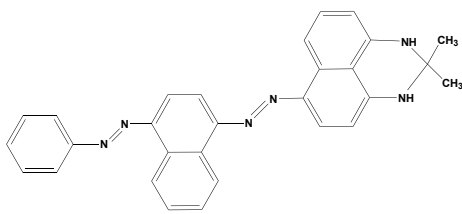
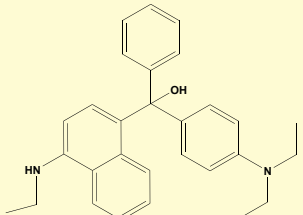
The following dyes can be examined individually and in mixtures.

Note: most dyes have a number of common names (they are available from the major chemical suppliers (eg, Sigma-Aldrich)). The commercial products are rarely pure (ie, you may see more than one spot on the chromatogram).

Avoid skin contact with dyes and their solutions since some are toxic.

Name(s)	Structure
Ceres yellow GGN, solvent yellow 56 (N,N-diethyl-4-(phenylazo)benzenamine)	
Quinoline yellow, solvent yellow 33 , Kaya-set yellow 2G (2-(2-quinolinyl)-1H-indene-1,3-(2H)-dione)	
Sudan yellow 3G, solvent yellow 16, disperse yellow (3-methyl-1-phenyl-4-(phenyldiazenyl)-1H-pyrazol-5-(4H)-one)	
Sudan green 4B, solvent green 3, ceres green BB (1,4-bis[(4-methylphenyl)amino]-9,10-anthracenedione)	
Oracet red G, disperse red 9 (1-(methylamino)anthraquinone)	
Sudan red 7B, fat red 7B (N-ethyl-1-((4-phenyldiazenyl)phenyldiazenyl)naphthalene-2-amine)	
Oil blue A, solvent blue 36, oil blue G (1,4-bis(isopropylamino)anthraquinone)	

Separation of Dyes (continued)

Name(s)	Structure
Oil blue 35, solvent blue 35 (1,4-bis(butylamino)anthraquinone)	
Sudan black, ceres black BN, solvent black 3 ((2,2-dimethyl-1,3-dihydroperimidin-6-yl)-4-phenylazo-1-naphthyl)diazene)	
Victoria blue BO, basic blue 7 (α,α -bis[4-(diethylamino)phenyl]-4-(ethylamino)naphthalene-1-methanol)	

Between them, these dyes have a variety of commercial applications, eg,

Biological stains

Colouring smoke

Fuel dyes

Ink dyes

Laser toners

Solvent, oil and fats dyes

Food dyes

Intermediates for making other dyes

Hair dyes

5. Essential oils (oils of plant origin).

Examine oils (as 1% solutions in dichloromethane): eg, pine oil, sweet bay oil (from *Laurus nobilis*), caraway seed oil, spearmint oil, eucalyptus oil, lavender oil, clove oil, orange peel oil.

Identify the major constituents of the oils using samples of: eg, 1,8-cineol ($\sim R_f = 0.45$), eugenol ($\sim R_f = 0.50$), methyl eugenol ($\sim R_f = 0.55$), camphene ($\sim R_f = 0.70$), terpinyl acetate ($\sim R_f = 0.57$), terpineol ($\sim R_f = 0.20$), linalol ($\sim R_f = 0.40$), limonene ($\sim R_f = 0.75$), citral ($\sim R_f = 0.60$), geraniol ($\sim R_f = 0.40$), cuminaldehyde ($\sim R_f = 0.50$), pinene ($\sim R_f = 0.80$), linalyl acetate ($\sim R_f = 0.65$), menthol ($\sim R_f = 0.35$), carvone ($\sim R_f = 0.54$). These would be made up as 1% solutions in dichloromethane and run on chromatography film alongside the essential oil sample. These approximate R_f values assume chromatography conditions similar to those given below.

Tlc film: silica gel.

Eluting solvent: eg, methylbenzene: ethyl ethanoate (92:8 by vol).

Locating agent (spray reagent): Try these:

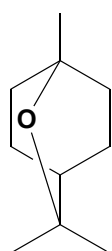
(i) vanillin (5g) in 50 cm³ ethanoic acid plus 1 cm³ concentrated sulphuric acid (**CARE**: add slowly). After spraying with this reagent (*in a well ventilated fume cupboard*) heat the chromatogram at about 100°C for a few minutes. Observe blue or purple spots on a white or pinkish background.

(ii) Anisaldehyde (1.5g) in 80 cm³ ethanol, 4 cm³ of concentrated sulphuric acid (**CARE**: add slowly) and 1 cm³ ethanoic acid. After spraying with this reagent (*in a well ventilated fume cupboard*) heat the chromatogram at about 100°C for a few minutes. Observe bluish or purple spots on a pinkish background.

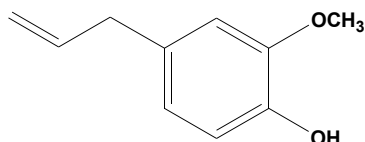
(iv) Dodecamolybdophosphoric acid (6g) in 40cm³ of ethanol. Spray in a well ventilated fume cupboard. Heat the sprayed chromatogram at 120°C for a couple of mins. Observe blue spots on a greenish background.

(v) CAM (cerium-ammonium-molybdate): ammonium pentamolybdate (4g), cerium(IV) sulphate (0.2g) in 80 cm³ of dilute sulphuric acid (2M). Spray in a well ventilated fume cupboard. Heat the sprayed chromatogram at about 120°C for a couple of mins. Dark blue spots on a pale yellowish background.

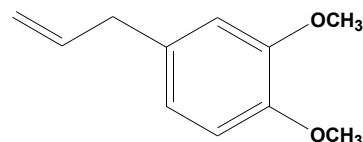
Structures of some essential oil components:



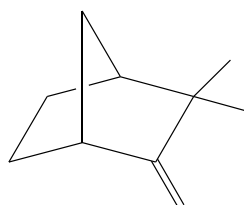
1,8-cineol
(present in bay oil)



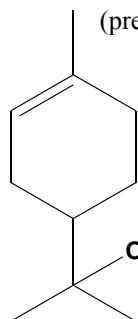
eugenol
(present in clove oil)



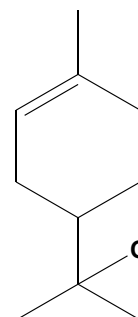
methyl eugenol (present in clove oil and cinnamon oil)



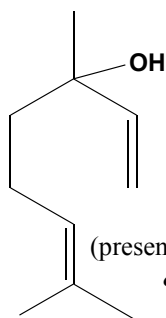
camphene
(present in Ceylon citronella oil)



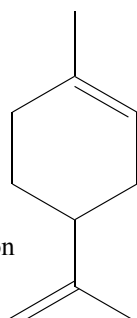
α-terpinyl acetate
(present in cardamon & cajuput oils)



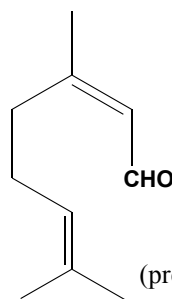
α-terpineol
(present in cajuput, pine & bay oils)



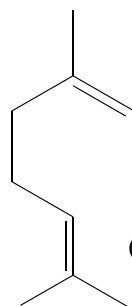
linalol
(present in mint, cinnamon & laurel oils)



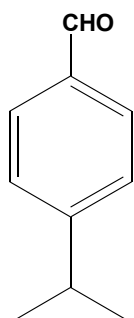
limonene
(present in orange peel & released by the cotton plant)



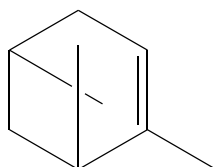
citral
(present in lemongrass & lemon myrtle)



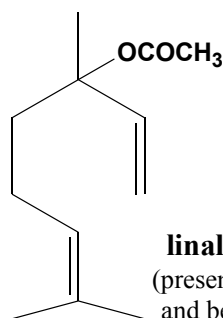
geraniol
(present in bergamot lavender, rose oils)



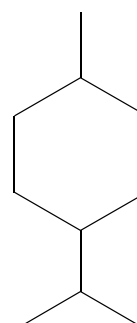
cuminaldehyde
(present in cumin seed, eucalyptus oils)



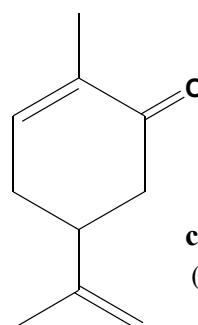
α-pinene
(present in oil from pine trees)



linalyl acetate
(present in lavender, and bergamot oils)



menthol
(present in mint oils)



carvone
(present in caraway and dill oils)